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HARD MAGNETIC PROPERTIES OF MULTILAYERED SmCo/Co PERMANENT MAGNETS

R. Andreescu and M. J. O'Shea
Department of Physics, Kansas State University, Manhattan KS 66506-2601

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We have prepared (Sm-Co)/Co multilayers with two different Co thicknesses to study the hard magnetic properties (coercivity H_{ci} and energy product BH_{max}) of nanostructured SmCo magnets consisting of hard SmCo and softer higher moment Co phases. Samples are annealed at a temperature in the range 450 – 700°C, for 20 minutes in vacuum (standard anneal) or rapidly annealed for 30 seconds under flowing nitrogen (rapid anneal) to form the hard Sm-Co phase. We find that samples subjected to a standard anneal at 500°C or a rapid anneal at 650°C show the largest energy products in these thin films, about 12 MG.Oe. This is closely related to the fact that the high field magnetization is highest for these anneal temperatures and decreases at higher anneal temperatures.

Sm-Co based materials have been investigated for many years for applications as hard permanent magnets. In particular permanent magnets based on these materials may be used at elevated temperatures, as high as 700°C [1,2]. It has been known for some time that a particular micro- or nanostructure is required to obtain the hard magnetic properties in these materials. Important aspects of this structure involve the size and shape of crystalline grains, their crystal orientation, and their inter-grain coupling. For sufficiently large coercivity ($H_{ci} > 2\pi M_s$) the value of the energy product BH_{max} is determined by M_s only and has an upper limit of $(2\pi M_s)^2$.

Thus most recent research to increase BH_{max} in permanent magnetic has focused on increasing M_s. An approach that has worked well is to make a nanostructured material composed of a hard magnetic phase and a high magnetic moment (usually soft) phase. These phases are exchange coupled so that the soft phase does not reverse in a small reverse applied field. These composite materials have larger values of M_s than the hard material by itself and thus have a larger energy product too. The hard and soft grains must have size of order 20 nm to be completely exchange coupled [3-7]. Several NdFeB/Fe [8], SmCo/Co [9,10] and SmCo/Fe [10] multilayers have been studied to understand the exchange coupling in nanostructured hard-soft materials and to try to increase the energy product.

In this work we look at the effect of (i) changing individual layer thickness in (Sm-Co)/Co multilayers, (ii) varying post-deposition anneal temperature and (iii) varying anneal time on the hard magnetic properties of SmCo/Co multilayers. We have already reported some work on a rapid anneal of NdFeB films [11].

We have prepared thin multilayered permanent magnets of the form 10x[$SmCo_5$(24 nm)/Co(d nm)] on a Si(100) substrate. The SmCo and Co layers are deposited by sputtering from a composite Sm-Co target and a separate Co target. Each of these samples is capped with a layer of Nb for protection. PIXE (proton induced x-ray emission) elemental analysis was done on a 500 nm thick single film of homogeneous Sm-Co deposited from the Sm-Co target and its composition is found to be $SmCo_{5.1}$ with an error of ±1%. After deposition films are annealed under vacuum for 20 minutes

(standard anneal) or are subjected to a rapid thermal anneal (rapid anneal) for 30 seconds under flowing nitrogen.

We find no preferential orientation of the crystalline phases formed after annealing. The phases we find in x-ray diffraction after an anneal include elemental hexagonal Co and a tentative identification of Sm_2Co_{17}. Crystallite size as determined from x-ray peak width is in the range 20 – 50 nm.

Magnetic properties are investigated using an MPMS5 SQUID magnetometer. In the as-deposited materials we find the room temperature coercivity increases from 0.16 kOe to 0.60 kOe when d, the Co layer thickness, is decreased from 10 nm to 6 nm. This result has also been seen by others [12].

On annealing at a sufficiently high temperature the coercivity increases significantly. A room temperature (300 K) hysteresis loop is shown in Figure 1 for a rapidly annealed sample with the magnetic field in the plane of the sample. We have also measured samples with the field perpendicular to the plane and find the differences in these hysteresis loops can be accounted for by demagnetization effects. Thus there is no in-plane or perpendicular anisotropy in these samples aside from that due to shape.

We find that hard magnetic properties depend strongly on anneal time, and Co layer thickness, d, in addition to the anneal temperature. Figure 2 shows coercivity H_{ci}, high field magnetization M and the energy product BH_{max} as a function of anneal temperature, all measured at room temperature (300 K) for the sample with d of 10 nm. As can be seen the anneal temperature above which the coercivity has a large value is 450°C for the sample subjected to standard anneal and is 600°C for the sample subjected to a rapid anneal. Kinematically this result makes physical

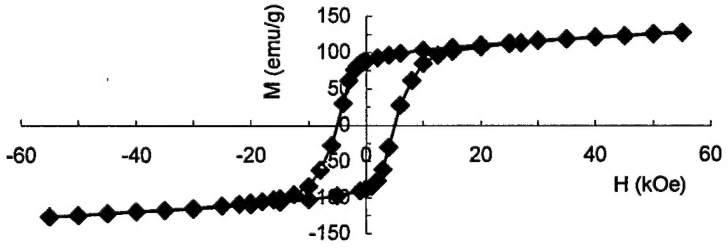

Figure 1. A hysteresis loop at room temperature for the sample with d of 10 nm after a rapid (30 second) anneal at 650°C. The applied field is in the plane of the sample.

sense since at shorter anneal times one would expect a higher temperature would be required to allow similar atomic rearrangements associated with crystallization to occur. The maximum coercivity for the rapidly annealed sample is considerably smaller than for the standard annealed sample while both the rapid and standard annealed sample have similar values for the maximum of the energy product.

Both the high field magnetization and energy product as a function of anneal temperature for the rapidly annealed sample are shifted by +150°C in anneal temperature compared to those for the standard annealed sample.

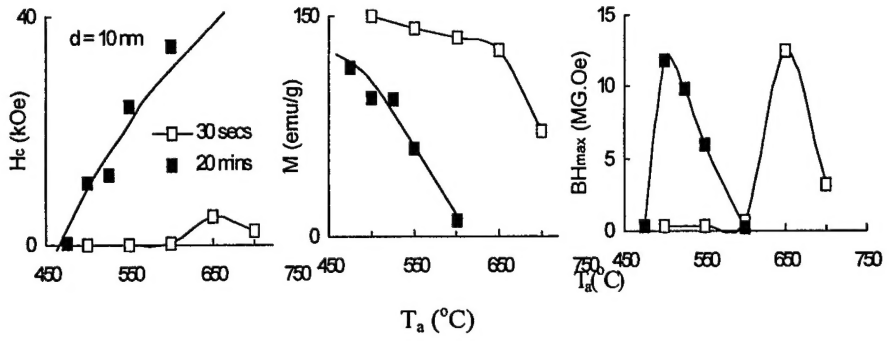

Figure 2. Coercivity H_{ci}, high field magnetization M and the energy product BH_{max}, measured at room temperature (300 K) for the sample with d of 10 nm after either a rapid (30 second) or a standard (20 minute) anneal.

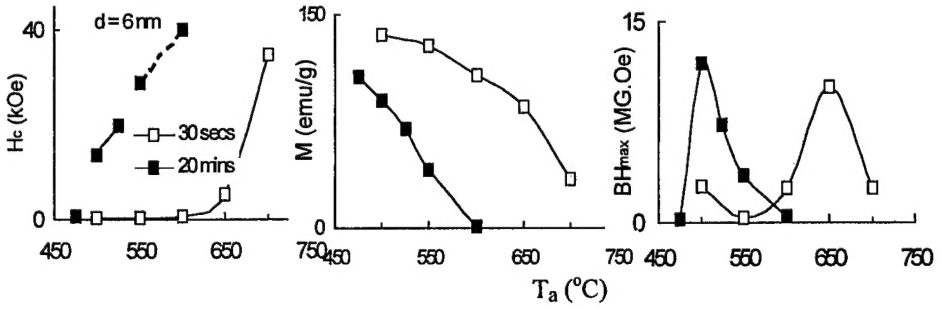

Figure 3. Coercivity H_{ci}, high field magnetization M and the energy product BH_{max}, measured at room temperature (300 K) for the sample with d of 6 nm after either a rapid (30 second) or a standard (20 minute) anneal.

Figure 3 shows coercivity H_{ci}, high field magnetization M and the energy product BH_{max} as a function of anneal temperature, all measured at room temperature (300 K) for the sample with d of 6 nm. The magnetization and energy product dependencies on anneal temperature and time are similar to those for the sample with d of 10 nm shown in Figure 2. There are some differences in the coercivites for these two values of d. Generally it appears that the smaller d sample tends to have larger coercivity for both the standard anneal and the rapid anneal. The samples with d of 10 nm also have a slightly larger high field magnetization.

We obtain our largest energy products in materials with coercivity of 4-12 kOe where the magnetization is large as can be seen from Figures 2 and 3. It is clear why the energy product shows a maximum. If the anneal temperature is too low, the hard Sm-Co phase does not form and the coercivity is low, < 1 kOe. If the anneal temperature is too high the magnetization shows a substantial decrease.

In Figure 4 we have collected together our results for coercivity as a function of anneal time for the sample with d of 10 nm annealed at 500°C. The energy product increases with anneal time for the chosen anneal temperature and layer thickness showing the importance of anneal time in these materials for a good energy product. The coercivity also increases with anneal time over the range of anneal times studied. Clearly too short an anneal time would not allow time for the sample to crystallize.

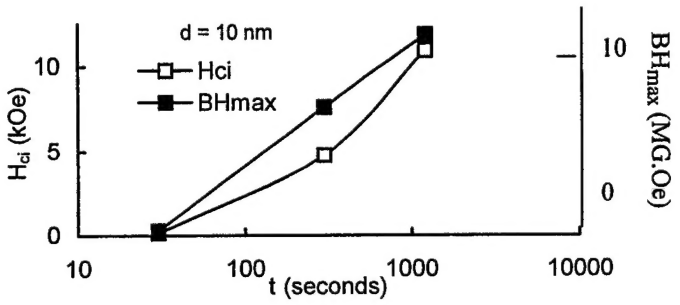

Figure 4. Coercivity H_{ci} and energy product BH_{max} as a function of anneal time for the sample with d of 10 nm at room temperature after an anneal at 500°C.

Thus anneal time and anneal temperature have a strong influence on hard magnetic properties. Increasing Co layer thickness (over our range of 6-10 nm) only increases the high field magnetization slightly. We are now looking at the structure of these materials to determine in detail the structural differences between the samples subjected to different anneal times.

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